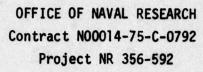
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Technical Report No. 1

ELECTROPOLYMERIZATION ON CARBON FIBERS-EFFECTS ON COMPOSITE PROPERTIES

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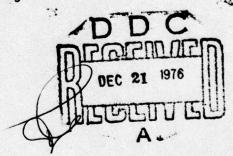
R.V. Subramanian and James J. Jakubowski

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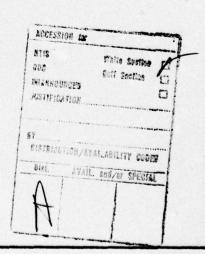
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Techniques were developed first for producing thin polymer coatings on carbon fibers by using the latter as electrodes in an electrolytic cell containing the monomer. A variety of vinyl and cyclic monomers having different types of functional groups were identified as capable of producing the desired polymer layer on the fibers under appropriate polymerization conditions. It was shown that, in many instances, the surface polymer was chemically bonded, i.e., grafted, to the carbon fiber.

Composite specimens were prepared by the incorporation of the coated fibers in an epoxy matrix. It was demonstrated that, in fact, the effect of the surface treatment on the interfacial properties of the resulting composite was manifested in variations of the measured interlaminar shear and impact strengths of the composite specimens. The increase in interlaminar shear was considered to be the result of improved fiber-matrix adhesion when compared to composite specimens prepared from untreated carbon fibers. The corresponding reversed trend in impact strength values was bened attributed to the effect of excessive fiber-matrix adhesion in causing brittle failure of the composite. (It was significant, therefore, when in exception to this general trend, it was also found that both interlaminar shear and impact strengths could simultaneously be increased. Implicit in this finding is the contribution of the electrochemically formed interlayer to one or more of the toughening mechanisms that are available to fiber reinforced composites. The potential value of interface modification by electrochemical polymerization in investigations of reinforcement mechanisms in carbon fiber composites is thus clearly indicated.



ELECTROPOLYMERIZATION ON CARBON FIBERS-EFFECT ON COMPOSITE PROPERTIES

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INTRODUCTION

Various methods of surface modification of carbon fibers have been tried in order to increase the interfacial bonding and interlaminar shear strength of carbon fiber-resin composites (1,2). Surface treatments that are now available to increase the adhesion of carbon fiber to the polymer matrix invariably lower the impact resistance of the composite. No theoretical route is available which maximizes impact strength and other properties also.

Interface tailoring seems to be the most promising approach to maximize the impact strength and fracture toughness while retaining the required optimum levels of other properties. By suitably forming and controlling the properties of a resin interlayer between reinforcement and matrix, it should be possible to modify crack initiation and propagation in the composite. An important development in composites research is the recent discovery that the structural properties of composite systems can be markedly changed through such incorporation of a resin interlayer between the matrix resin and the reinforcement (3-7).

Electrochemical polymerization seemed to be an attractive process for forming a polymer layer of controlled thickness and properties on carbon fibers before their incorporation in a resin matrix. The present research program involves an investigation of the electropolymerization of monomers on carbon fiber electrode surfaces to form a polymer layer prior to incorporating the fibers in composites and a study of the effects of such surface modification of the fibers on the properties of polymer composites made from them.

EXPERIMENTAL

Carbon fibers were used as electrodes in the center compartment of a 3-compartment cell, containing solvent, electrolyte, and monomer. Platinum electrodes were placed on each of the two end compartments along with solvent and electrolyte. Electropolymerizations were conducted at constant DC voltage.

Composite specimens were prepared from Hercules type AU carbon fiber tow. The tow was wound on H-type frames and exposed to an electropolymerization treatment, after which the fibers were quickly removed, rinsed and vacuum dried at 50°C for 12 hours. An epoxy resin, EPON 828 with meta-phenylene diamine at 100:14 parts by weight, was used as the matrix resin to prepare composites from the coated fibers.

Interlaminar shear strength was measured using flat beam specimens as described in ASTM D2344-72 using a span to thickness ratio of 4 to 1 and a crosshead speed of 1.0mm/min. For each electropolymerization treatment, the shear strength was measured at three fiber volume fractions between 45 and 75%.

Similarly, the impact strength of composites prepared from fibers which had undergone an electropolymerization treatment were also measured. Impact specimens were tested in accordance with ASTM D256-73, method A, Izod. The best straight line through the plot of impact strength against fiber volume fraction of measurements taken at 9 different fiber volume fractions was obtained.

RESULTS AND DISCUSSION

A variety of monomer-solvent-electrolyte systems were studied to determine the conditions necessary for forming a polymer coating on carbon fibers by electropolymerization. Several commercially available carbon fibers having different mechanical properties (Thornel, Fortafil and Hercules) were used as electrodes. It became apparent that carbon fibers were a very good substrate for electropolymerization. Polymer coatings, many visually observable, formed quickly within seconds after application of current, with no apparent differences observed between fibers obtained from different manufacturers.

Polymer presence was observed by weight increases of the fibers after polymerization, scanning electron micrographs of the polymer coatings, and, when possible, infrared spectral analysis of polymer extracted from the fibers. Typical systems in which polymer coatings were observed are shown in Table I.

It was noted that measurements of the drop in cell current during electrolysis proved to be an inaccurate method of detecting and following the formation of a polymer coating on the fibers. While some systems like diacetoneacrylamide and VTBN had significant current drops during polymerization, many systems did not have any detectable current drops and some even had a slight increase in cell current. Also it was observed that small current fluctuations were present in some systems, such as the sulphuric-acid-water solvent-electrolyte system, even when monomers were absent. Therefore, although very qualitatively useful, current changes during electrolysis were not used as anything more than tentative indications of the presence or absence of a polymer coating, confirmation for which was obtained only from the other methods listed above.

The major step in conducting these polymerizations appears to be the selection of a solvent-electrolyte system which is capable of forming a solution with the monomer and which has sufficient current conducting properties. Dimethyl formamide and dimethyl sulfoxide proved very useful in this respect as solvents. Both homo- and copolymerizations in aqueous and non-aqueous solvent systems were observed to form coatings. It can readily be seen, from Table I, that monomers containing a variety of functional groups, terminal vinyl, carboxylic acid, anhydride, epoxy, and aziridinyl, were observed to form coatings. It is interesting that even a liquid copolymer, VTBN (vinyl terminated butadiene acrylonitrile), could be further polymerized by this technique.

Polymerizations conducted so far have produced polymer coatings with a wide range of chemical and mechanical properties which could be used to modify the carbon fiber-polymer matrix interface of composites prepared from fibers treated by electropolymerization. A flexible polymer interface could be introduced using systems such as methyl acrylate or VTBN, while a stiff interface could be achieved by the styrene or styrene-acrylonitrile systems. Methyl methacrylate and styrene monomers would be expected to yield linear uncrosslinked polymer coatings while monomers which are difunctional, such as N,N'-methylene-bis-acrylamide, PFAZ 300, and EPON 828 would be expected to crosslink in the systems they were used in.

Chemically binding the matrix resin to the electropoly-merized interface could be achieved by residual, epoxy, carboxylic acid or aziridinyl functional groups present in the coating after electropolymerization. Acrylic acid, Epon 828, or glycidyl methacrylate are examples of monomers that could be used for this purpose. Such a possibility is particularly interesting if the polymer coating formed initially in electropolymerization is chemically bonded, i.e., grafted to the carbon fiber surface. In such cases, the polymer coating can act in the manner of silane coupling agents used in glass fiber composites to bridge the fiber-matrix interface.

It was therefore significant that grafting of the coating polymer was found to be a common occurrence in many, though not all, monomer systems evaluated. Fortafil 3T and 5T untreated carbon fibers were subjected to electropolymerization followed by continuous extraction for a period of 120 hours to insure removal of all the unbonded polymer which would be soluble. Observed weight increases were used as preliminary evidence of the presence of a grafted polymer. Electron micrographs, taken at 1600X, confirmed the presence of grafting. When grafting occurred, it was observed with both high modulus, 5T, and low modulus, 3T carbon fibers. From among many systems studied one could see that diacetone acrylamide, methyl methacrylate and styrene did form graft polymers on graphite fibers.

The mechanism of grafting was not investigated. Organic functional groups such as -COOH and -OH, are present on carbon fibers. These groups are capable of forming free radical sites, for example, by chain transfer. Initiation of free radical polymerization, or, termination by combination of growing polymer radicals at these sites could lead to the observed polymer grafting.

COMPOSITES OF COATED FIBERS

The interlaminar shear and impact strengths of epoxy polymer composites prepared from Hercules type AU carbon fibers coated by electropolymerization of a series of different monomer systems were measured. Systems were selected to include representatives of the various type of monomers, in both

aqueous and nonaqueous solvent-electrolyte systems, encountered during the screening process.

Composites prepared from Hercules type AU, untreated, and type AS, commercially surface treated, carbon fibers without further treatment were also tested for comparison.

Incorporation of a polymer interlayer on carbon fibers prior to imbedding into a polymer matrix was found to significantly affect the interlaminar shear and impact strengths of the composite. Although further detailed studies are needed to standardize the electropolymerization technique in order to obtain optimum results, a number of experimental parameters have been identified. The resulting composite mechanical properties were found to be a function of the monomer, solvent, polymerization time, fiber content, and post electropolymerization treatment of the coated carbon fibers. Using different types of monomers, vinyl, epoxy, etc., gave rise to significantly different shear strengths keeping all other parameters constant. (See Fig. 1, data points omitted for clarity). The use of water as a solvent appears to be superior in improving shear strength over organic solvent systems and consistently improved the shear strength over the whole range of fiber contents tested when compared to composites prepared from untreated fiber.

Mechanical properties are sensitive to the polymerization time, as was shown in the DAA system. Shear and impact properties were shown to vary with polymerization time. The effect of polymerization time on mechanical properties was also dependent on composite fiber content. In most cases, for a particular polymerization time, an increasing trend of shear strength was observed with increasing fiber content. The best example of this trend is shown in the PFAZ 300 system, (Fig. 2), where nearly a 100% increase in shear strength was observed over the range of fiber contents tested. In the DAA system it was found that a 2.5 second time (Fig. 2) gave rise to an increasing trend in shear strength while a 10 second time (Fig.3) reversed this trend to a decreasing one over the 45 to 75 volume percent fiber range.

Similar trends with respect to fiber content were also observed in the impact tests, (Fig. 4; data points omitted for clarity). Different electropolymerization times in the DAA system, viz., 2.5 and 10 seconds (Fig. 4) gave rise to decreasing and increasing trends, respectively, in the impact strength variation with increasing fiber content. When the trends of impact strength are compared to trends of shear strength, it is seen that the DAA system yielded an increasing impact strength whenever decreasing shear strength is observed. Unlike the DAA system, PFAZ 300 (Fig. 4) caused an increasing trend in both shear and impact strength when fiber content was increased. The result of these trends was an improvement of both the shear and impact strengths of composites having a fiber volume fraction between 65 to 75 percent compared to composites prepared from untreated fibers.

Finally, shear properties and most probably, also other mechanical properties of composites prepared from fibers which had undergone an electropolymerization treatment are sensitive to post-polymerization treatments prior to imbedding into a matrix. A thermal treatment of the fiber and DAA polymer coating significantly decreased the shear strength over the whole range of fiber contents measured (Fig. 3). Postreatments therefore can be expected to alter the mechanical properties of composites and merit careful investigation.

Experimental evidence presented here has demonstrated that electropolymerization techniques can be used to modify composite interfacial properties. Further work is presently directed towards optimization of this technique.

ACKNOWLEDGEMENTS

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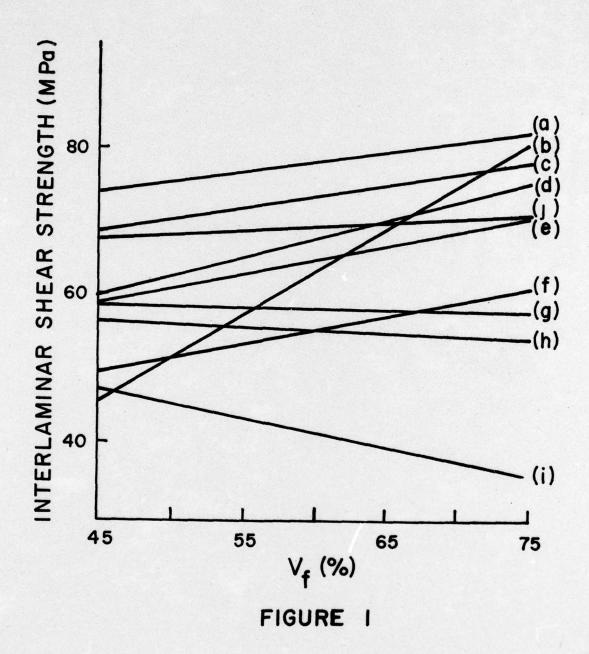
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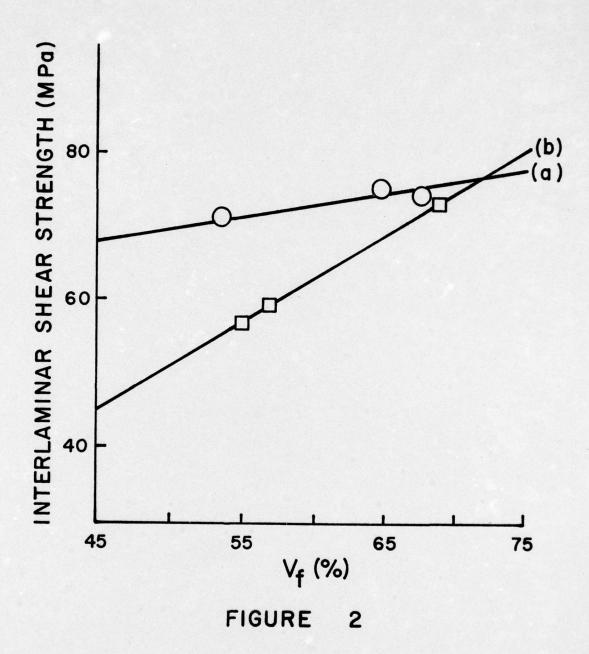
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- 7. M. Xanthos and R.T. Woodham, J. Appl. Polym. Sci., <u>16</u>,381 (1972).
- Fig. 1. Interlaminar Shear of Composites Prepared from Fibers Coated with (a) Acrylic Acid (b) PFAZ 300 (c) DAA (d) Styrene (e) MMA (f) Styrene/Acrylonitrile (g) ϵ -Caprolactam (h) EPON 828/Phthalic Anhydride (i) VTBN (j) and Untreated Hercules AU Carbon Fiber
- Fig. 2. Interlaminar Shear of Composites Prepared from Fibers Coated with (a) DAA (b) PFAZ 300
- Fig. 3. Interlaminar Shear of Composites Prepared from Fibers Coated with (a) DAA 10 seconds (b) DAA 10 Seconds, Annealed
- Fig. 4. Impact Strength of Composites Prepared from Fibers Coated with (a) PFAZ 300 (b) DAA 10 Seconds (c) DAA 2.5 seconds (d) Hercules Au and (e) Hercules AS Carbon Fiber

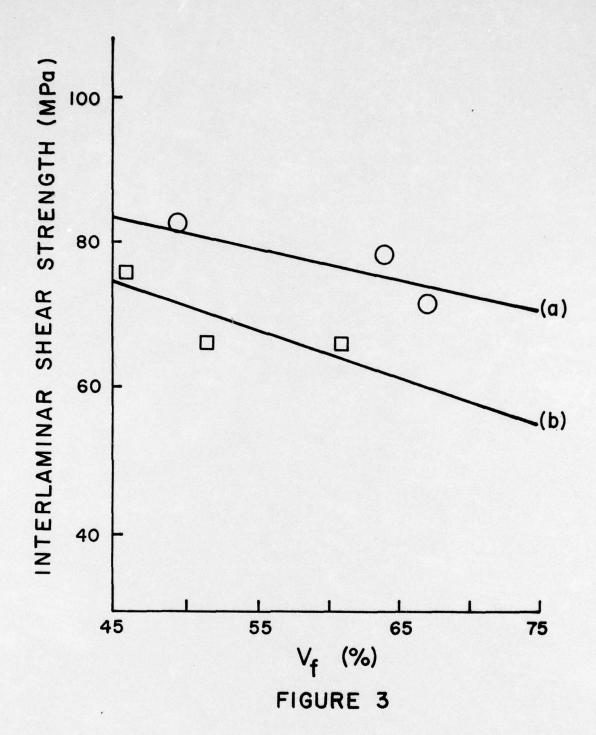
Table I. Electropolymerization Systems

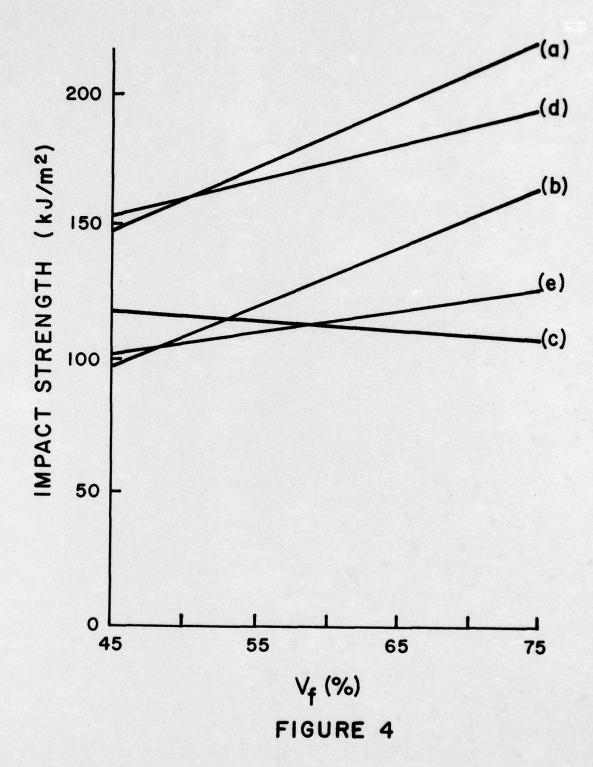
Monomer(s)	Solvent-Electrolyte	Locus	
0.5 <u>M</u> Diacetone Acrylamide	0.1 N H2504	Cathode	
1.5 <u>M</u> Acrylic Acid	0.1 N H2504	Anode	
1.5 <u>M</u> Acrylic Acid	0.1 N H2504	Cathode	
1.6 M Methyl Methacrylate	0.2 N NaNO3-DMF	Cathode	
3.1 <u>M</u> Methyl Methacrylate	0.2 N NaNO3-DMSO	Anode	
5.5 <u>M</u> Methyl Acrylate	0.2 N LiOAc-EtOH	Cathode	
2.5 <u>M</u> Styrene	0.2 N NaNO3-DMF	Cathode	
1.1 <u>M</u> ε-Caprolactam	0.2 N NaNO3-DMF	Anode	
Acrylonitrile	Sat'd (CH ₃)4NC1	Anode	
3.1 <u>M</u> Vinyl Acetate	0.2 N LiOAc-CH3OH	Anode	
0.04 g/ml Hycar VTBN	0.2 N NaNO3-DMF	Cathode	
0.05 g/m1 PFAZ 300	0.2 N NaNO3-DMF	Cathode	
1.6 M EPON 828 0.4 M Phthalic Anhydride	0.2 <u>N</u> LiC1-DMF	Anode	
0.8 <u>M</u> Styrene 0.8 <u>M</u> Acrylonitrile	0.3 N ZnC12-CH2C12	Cathode	
2.4 M Glycidyl Methacrylate 1.6 M Phthalic Anhydride	0.2 <u>N</u> LiC1-DMF	Anode	
1.1 M Glycidyl Methacrylate 2.1 M Acrylic Acid	0.2 <u>N</u> LiOAc-EtOH	Anode	
1.4 M Methyl Methacrylate 0.7 M Acrylic Acid	0.2 <u>N</u> LiOAc-EtOH	Anode	
2.6 M Methyl Acrylate 2.9 M Acrylic Acid	0.2 <u>N</u> LiOAc-EtOH	Anode	
0.9 M Methyl Methacrylate 1.4 M Acrylic Acid	0.2 <u>N</u> LiOAc-EtOH	Anode	

Polymerizations at 7-12 volts DC.









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